

TestAmerica Laboratories, Inc.

ANALYTICAL REPORT

Perfluorocarbon (PFC) Analysis

Lot #: D9K130498

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Case Narrative

TestAmerica Denver utilizes USEPA approved methods in all analytical work. The samples presented in this report were analyzed for the parameters listed on the methods summary page in accordance with the methods indicated. Dilution factors and footnotes are provided on each datasheet to assist in the interpretation of the results.

The results relate only to the samples in this report and meet all requirements of NELAC. All data have been reviewed for compliance with the laboratory QA/QC plan and have found to be compliant with laboratory protocols with any exceptions noted below.

Please note that Non-Detect (ND) results have been evaluated down to the Method Detection Limit (MDL) and should be considered ND at the MDL. Unless otherwise noted, results for solids have been dry weight corrected.

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Sample Arrival and Receipt

The following report contains the analytical results for four samples received at TestAmerica Denver on November 13, 2009, according to documented sample acceptance procedures. The samples were received in good condition at a temperature of 2.3°C.

The samples were received after the laboratory prescribed holding time had expired for PFC and FOSA analyses. The client was notified on November 13, 2009.

The sample collection times were listed as 12-12 on the chain-of-custody. The client informed the laboratory on November 16, 2009, the samples are composites that were collected from 12 AM to 12 AM on the respective days. Due to a limitation in the LIM system, the times had to be entered as 12:01 AM.

No other anomalies were encountered during sample receipt.

Standards

Analytical standards were prepared using commercially available certified solutions containing all compounds of interest.

The mass labeled compounds 13C4 PFBA, 13C2 PFHxA, 18O2 PFHxS, 13C4 PFOA, 13C4 PFOS, 13C5 PFNA, 13C2 PFDA, 13C2 PFUnA, 13C2 PFDoA, and D3 MeFOSA were introduced at the extraction step and were used for internal standards for the quantitation of the target compounds.

Sample Extraction and Analysis

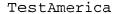
The samples presented in this report were extracted for the target analytes by TestAmerica Denver's Standard Operating Procedure (SOP) DV-OP-0019 and analyzed for the target analytes by TestAmerica Denver's SOP DV-LC-0012.

Method QC Samples

The Method Blank is processed reagent water spiked with internal standard and prepared with each batch of 20 samples of the same matrix. The method blanks were non-detect at the reporting limits for the target analytes.









Each batch is prepared with low and mid level Laboratory Control Samples (LCS). The LCS recoveries for both levels were within established control limits, with the exception of the items noted in section Analytical Comments.

Analytical Comments

The Standard Operating Procedure (SOP) was altered slightly in the sample preparation for FOSA. Sodium hydroxide was added to all four samples to obtain a pH of 14 instead of the SOP required <2. The basic pH is generating better internal standard recoveries for MeFOSA.

The organic preparation chemist noted two cartridges were required to extract samples I-3, I-4, and E-4 for FOSA.

Each sample is analyzed to achieve the lowest possible reporting limits within the constraints of the method. Due to matrix interference, all four samples had to be analyzed at dilutions. The reporting limits have been adjusted relative to the dilutions required. Please note the samples were dark brown or dark orange in color and cloudy.

All four samples were received at the laboratory after the laboratory prescribed holding time had expired. There is no prescribed regulatory holding time requirement for PFCs. The scientific literature indicates PFCs are highly persistent compounds in the environment. TestAmerica Denver has conducted stability studies indicating medium- and low-level standard solutions of PFOA are stable for at least three months in glass, polystyrene, and polypropylene plastics at 4±2 °C. The 7-day/40-day and 14-day/40-day holding times listed above are based on the general EPA convention for the holding time of extractable organic compounds in water and soil. Please note the sample results should be considered estimated.

Samples I-3, I-4, and E-4 exhibited internal standard recoveries outside the QC control limits. These anomalies are due to matrix interferences; therefore, corrective action is deemed unnecessary.

Due to a limitation in the LIMS system, the low-level LCS associated with QC batch 9320505 reported the percent recoveries for two PFCs as 0.0%. These compounds were recovered within the control limits, as outlined below.

Compound	Low-Level LCS Actual Recovery	Control Limits	Low-Level LCS Actual Result	MDL
PFTriA	71%	44-164%	0.0142 ug/L	0.01772 ug/L
PFTeA	67%	47-172%	0.0134 ug/L	0.01456 ug/L

As the compounds were detected below the Method Detection Limits (MDL), the system reports the percent recoveries as 0.0%.

The mid-level LCS/LCSD and low-level LCS associated with QC batch 9320512 exhibited percent recoveries above the QC control limits for Perfluorooctane sulfonamide (FOSA). This is an indicator that data may be biased high. As no detectable concentrations are present in the associated samples, corrective action is deemed unnecessary.

The method required MS/MSD could not be performed for QC batches 9320505 and 9320512, due to insufficient sample volume. Method precision and accuracy have been verified by the acceptable low-level LCS and mid-level LCS/LCSD analyses data.



TestAmerica

The closing Continuing Calibration Verification (CCV) standard associated with samples in QC batch 9320512, exhibited a %D value out of range, biased high, for Perfluorooctane sulfonamide (FOSA). This is an indicator that data may be biased high. As no detectable concentrations are present in the associated samples, corrective action is deemed unnecessary.

The Standard Operating Procedure (SOP) was altered slightly for these samples in the sample prep and LC conditions. The alterations are listed below.

Solvents are now the same as they were in the original SOP and run per the following gradient: From 0 to 11 minutes, the flow rate is 0.4 mL/minute and the MeOH ramps up from 25% to 100%. From 11 to 11.01 minutes, the flow rate increases to 0.7 mL/minute and this flow is diverted from the MS. At 13 minutes the flow rate decreases back down to 0.4 mL/minute and 25% MeOH. The column then equilibrates to 14 minutes.

PFTriA and PFTeA now use 13C2 PFUnA as their internal standard instead of 13C2 PFDoA.

No other anomalies were observed.





EXECUTIVE SUMMARY - Detection Highlights



PARAMETER	RESULT	REPORTING LIMIT	UNITS	ANALYTICAL METHOD
I-3 11/05/09 00:01 001				
Perfluorooctanesulfonate Perfluorohexanoic acid (PFHxA) Perfluorobutane sulfonate (PFB		0.20 0.20 0.20	ug/L ug/L ug/L	DEN -LC-0012 DEN -LC-0012 DEN -LC-0012
E-3 11/05/09 00:01 002				
Perfluorooctanoic Acid Perfluorooctanesulfonate Perfluorobutanoic acid (PFBA) Perfluoropentanoic acid (PFPA) Perfluorohexanoic acid (PFHxA) Perfluorodecanoic acid (PFDA) Perfluorobutane sulfonate (PFB I-4 11/05/09 00:01 003	0.37 0.17 J	0.20 0.20 0.20 0.30 0.20 0.20	ug/L ug/L ug/L ug/L ug/L ug/L	DEN -LC-0012
Perfluorooctanesulfonate Perfluorohexanoic acid (PFHxA) Perfluorobutane sulfonate (PFB E-4 11/05/09 00:01 004		0.20 0.20 0.20	ug/L ug/L ug/L	DEN -LC-0012 DEN -LC-0012 DEN -LC-0012
Perfluorooctanoic Acid Perfluorooctanesulfonate Perfluorobutanoic acid (PFBA) Perfluoropentanoic acid (PFPA) Perfluorohexanoic acid (PFHxA) Perfluorobutane sulfonate (PFB	0.30	0.20 0.20 0.20 0.30 0.20 0.20	ug/L ug/L ug/L ug/L ug/L ug/L	DEN -LC-0012 DEN -LC-0012 DEN -LC-0012 DEN -LC-0012 DEN -LC-0012 DEN -LC-0012



METHODS SUMMARY

D9K130498

PARAMETER ANALYTICAL PREPARATION METHOD METHOD

LC/MS/MS PFCs DEN -LC-0012 SW846 FOSA spec

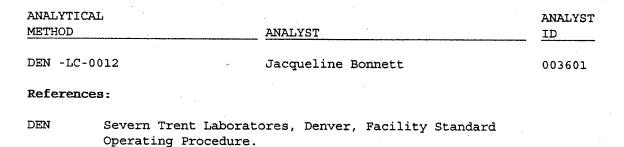
References:

DEN Severn Trent Laboratores, Denver, Facility Standard Operating Procedure.



METHOD / ANALYST SUMMARY

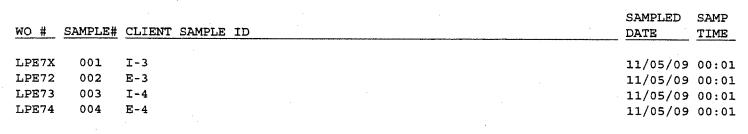






SAMPLE SUMMARY





NOTE(S):

- The analytical results of the samples listed above are presented on the following pages.
- All calculations are performed before rounding to avoid round-off errors in calculated results.
- Results noted as "ND" were not detected at or above the stated limit.
- This report must not be reproduced, except in full, without the written approval of the laboratory.
- Results for the following parameters are never reported on a dry weight basis: color, corrosivity, density, flashpoint, ignitability, layers, odor, paint filter test, pH, porosity pressure, reactivity, redox potential, specific gravity, spot tests, solids, solubility, temperature, viscosity, and weight.



Client Sample ID: I-3

HPLC

Lot-Sample #: D9K130498-0 Date Sampled: 11/05/09 00 Prep Date: 11/16/09 Prep Batch #: 9320505 Dilution Factor: 10		1: 11/13/09 2: 12/05/09	•	ix:	WATER
	Method	: DEN -LC-	-0012		
PARAMETER	RESULT	REPORTIN LIMIT	IG UNITS	MDL	
Perfluorooctanoic Acid	ND	0.20	uq/L	0.098	
Perfluorooctanesulfonate	0.22	0.20	ug/L	0.13	
	PERCENT	RECOVERY			

LIMITS

(60 - 155)

(45 - 130)

RECOVERY

114

102



SURROGATE

13C4 PFOA

13C4 PFOS

Client Sample ID: I-3

HPLC

Lot-Sample #: D9K130498-001	Work Order #: LPE7X2AA	Matrix WATER
-----------------------------	------------------------	--------------

 Date Sampled...:
 11/05/09 00:01 Date Received...:
 11/13/09

 Prep Date.....:
 11/16/09 Analysis Date...:
 12/23/09

 Prep Batch #...:
 9320505 Analysis Time...:
 22:12

Dilution Factor: 10

Method..... DEN -LC-0012

		REPORTING	3	
PARAMETER	RESULT	LIMIT	UNITS	MDL
Perfluorobutanoic acid (PFBA)	ND	0.20	ug/L	0.098
Perfluoropentanoic acid (PFPA)	ND	0.30	ug/L	0.11
Perfluorohexanoic acid (PFHxA)	0.15 J	0.20	ug/L	0.029
Perfluoroheptanoic acid (PFHpA)	ND	0.20	ug/L	0.13
Perfluorononanoic acid (PFNA)	ND	0.20	ug/L	0.17
Perfluorodecanoic acid (PFDA)	ND	0.20	ug/L	0.078
Perfluoroundecanoic acid (PFUn A)	ND	0.20	ug/L	0.069
Perfluorododecanoic acid (PFDo A)	ND	0.20	ug/L	0.15
Perfluorotridecanoic acid (PFT riA)	ND	0.20	ug/L	0.18
Perfluorotetradecanoic acid (P FTeA)	ND	0.20	ug/L	0.15
Perfluorobutane sulfonate (PFB S)	0.095 J	0.20	ug/L	0.082
Perfluorohexane sulfonate (PFH xS)	ND	0.30	ug/L	0.070
	PERCENT	RECOVERY		
SURROGATE	RECOVERY	LIMITS		
13C4 PFOA	186 *	(60 - 15!	 5)	
13C4 PFOS	142 *	(45 - 130		
13C4 PFBA	146 *	(36 - 130		
13C2 PFHxA	130	(55 - 135		
1802 PFHxS	135 *	(61 - 130		
13C5 PFNA	147 *	(54 - 132	•	
13C2 PFDA	176 *	(53 - 130		
13C2 PFUnA	180 *	(37 - 130		
13C2 PFDoA	190 *	(26 - 13)	•	
NOTE(S):				•

^{*} Surrogate recovery is outside stated control limits.



J Estimated result. Result is less than RL.

Client Sample ID: I-3

HPLC

Lot-Sample #...: D9K130498-001 Work Order #...: LPE7X1AD Matrix....: WATER Date Sampled...: 11/05/09 00:01 Date Received..: 11/13/09 **Prep Date....:** 11/16/09 Analysis Date..: 12/01/09 **Prep Batch #...:** 9320512 Analysis Time..: 18:03 Dilution Factor: 500 Method..... DEN -LC-0012 REPORTING PARAMETER RESULT UNITS Perfluorooctane sulfonamide (F 25 2.9 ug/L OSA)

RECOVERY

(37 - 130)

LIMITS

PERCENT

89

RECOVERY

SURROGATE

MeFOSA

Client Sample ID: E-3

HPLC

Lot-Sample #:	D9K130498-002	Work Order #:	LPE721AA	Matrix	WATER
Date Sampled:	11/05/09 00:01	Date Received:	11/13/09		
Pren Date .	11/16/09	Analysis Date .	12/05/09		

Prep Batch #...: 9320505 Analysis Time..: 01:33

Dilution Factor: 10

13C4 PFOS

Method.....: DEN -LC-0012

(45 - 130)

		REPORTING		
PARAMETER	RESULT	LIMIT	UNITS	MDL
Perfluorooctanoic Acid	0.27	0.20	ug/L	0.098
Perfluorooctanesulfonate	0.34	0.20	ug/L	0.13
	PERCENT	RECOVERY		
SURROGATE	RECOVERY	LIMITS	_	
13C4 PFOA	107	(60 - 155)		

92



Client Sample ID: B-3

HPLC

Lot-Sample #...: D9K130498-002 Work Order #...: LPE722AA Matrix..... WATER

 Date
 Sampled...:
 11/05/09
 00:01
 Date Received...:
 11/13/09

 Prep
 Date....:
 11/16/09
 Analysis Date...:
 12/23/09

 Prep
 Batch #...:
 9320505
 Analysis Time...:
 22:27

Dilution Factor: 10

Method..... DEN -LC-0012

		REPORTING		
	ESULT	LIMIT	UNITS	MDL
uorobutanoic acid (PFBA) 0	.19 J	0.20	ug/L	0.098
	.56	0.30	ug/L	0.11
uorohexanoic acid (PFHxA) 0	.37	0.20	ug/L	0.029
uoroheptanoic acid (PFHpA N	D	0.20	ug/L	0.13
uorononanoic acid (PFNA) N	D	0.20	ug/L	0.17
uorodecanoic acid (PFDA) 0	.17 J	0.20	ug/L	0.078
uoroundecanoic acid (PFUn N	D	0.20	ug/L	0.069
uorododecanoic acid (PFDo N	D .	0.20	ug/L	0.15
uorotridecanoic acid (PFT N A)	D	0.20	ug/L	0.18
uorotetradecanoic acid (P N eA)	D ,	0.20	ug/L	0.15
uorobutane sulfonate (PFB 0	.29	0.20	ug/L	0.082
lorohexane sulfonate (PFH N	D ·	0.30	ug/L	0.070
Pl	ERCENT	RECOVERY		
GATE RI	ECOVERY	LIMITS		
PFOA 1:	33	(60 - 155)	•	
	11	(45 - 130)		
	07	(36 - 130)		
	18	(55 - 135)		
	13	(61 - 130)		
	12	(54 - 132)		
	18	(53 - 130)		
	13	(37 - 130)		
· · · · · · · · · · · · · · · · · · ·		· ·		
PFDOA 1(5): continued result. Result is less than RL	00	(26 - 130)		

J Estimated result. Result is less than RL.



TestAmerica

Client Sample ID: E-3

Lot-Sample #: D9K130498-002	Work Order #:	LPE721AC	Matr	ix:	WATER
Date Sampled: 11/05/09 00:03	Date Received:	11/13/09			
Prep Date: 11/16/09	Analysis Date:	12/01/09			
Prep Batch #: 9320512	Analysis Time:	18:08			
Dilution Factor: 10				•	
	Method:	DEN -LC-00	12		
		REPORTING			
PARAMETER	RESULT	LIMIT	UNITS	MDL	
Perfluorooctane sulfonamide (F	ND	0.50	ug/L	0.057	
OSA)					
	PERCENT	RECOVERY			
SURROGATE	RECOVERY	LIMITS			
MeFOSA	90	(37 - 130)			



Client Sample ID: I-4

HPLC

Lot-Sample #: D9K130498-003 Date Sampled: 11/05/09 00:01 Prep Date: 11/16/09 Prep Batch #: 9320505 Dilution Factor: 10	==	11/13/09 12/05/09	Matrix	WATER
Dilucion Factor: 10	Method:	DEN -LC-00	12	
PARAMETER	RESULT	REPORTING LIMIT	UNITS	MDL
Perfluorooctanoic Acid	ND	0.20	ug/L	0.098
Perfluorooctanesulfonate	0.27	0.20	ug/L	0.13
	PERCENT	RECOVERY		
SURROGATE	RECOVERY	LIMITS		
13C4 PFOA	104	(60 - 155)		

95

(45 - 130)



13C4 PFOS

Client Sample ID: I-4

HPLC

Lot-Sample #:	D9K130498-003	Work Order #:	LPE732AA	Matrix:	WATER
Date Sampled:	11/05/09 00:01	Date Received:	11/13/09		
Prep Date:	11/16/09	Analysis Date:	12/23/09		
Prep Batch #:	9320505	Analysis Time:	22:42		

Prep Batch #...: 9320505 Dilution Factor: 10

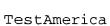
	Method:	DEN -LC-00	12	•
· · · · · · · · · · · · · · · · · · ·		REPORTING		
PARAMETER	RESULT	LIMIT	UNITS	MDL MDL
Perfluorobutanoic acid (PFBA)	ND	0.20	ug/L	0.098
Perfluoropentanoic acid (PFPA)	ND	0.30	ug/L	0.11
Perfluorohexanoic acid (PFHxA)	0.11 J	0.20	ug/L	0.029
Perfluoroheptanoic acid (PFHpA)	ND	0.20	ug/L	0.13
Perfluorononanoic acid (PFNA)	ND	0.20	ug/L	0.17
Perfluorodecanoic acid (PFDA)	ND	0.20	ug/L	0.078
Perfluoroundecanoic acid (PFUn A)	ND	0.20	ug/L	0.069
Perfluorododecanoic acid (PFDo A)	ND	0.20	ug/L	0.15
Perfluorotridecanoic acid (PFT riA)	ND	0.20	ug/L	0.18
Perfluorotetradecanoic acid (P FTeA)	ND	0.20	ug/L	0.15
Perfluorobutane sulfonate (PFB S)	0.59	0.20	ug/L	0.082
Perfluorohexane sulfonate (PFH xS)	ND	0.30	ug/L	0.070
•	PERCENT	RECOVERY		
SURROGATE	RECOVERY	LIMITS		
13C4 PFOA	165 *	(60 - 155)		
13C4 PFOS	115	(45 - 130)		
13C4 PFBA	129	(36 - 130)		
13C2 PFHxA	119	(55 - 135)		
1802 PFHxS	116	(61 - 130)		
13C5 PFNA	127	(54 - 132)		•
13C2 PFDA	145 *	(53 - 130)		2.5
13C2 PFUnA	159 *	(37 - 130)		
	165 *	(26 - 130)		

^{*} Surrogate recovery is outside stated control limits.

J Estimated result. Result is less than RL.

Client Sample ID: I-4

Lot-Sample #: D9K130498-003	Work Order #:		Mat	WATER		
Date Sampled: 11/05/09 00:01	Date Received:	11/13/09				
Prep Date: 11/16/09	Analysis Date:	12/01/09				
Prep Batch #: 9320512	Analysis Time:	18:13				
Dilution Factor: 500						
	Method:	.: DEN -LC-0012				
•		REPORTING				
PARAMETER	RESULT	LIMIT	UNITS	MDL		
Perfluorooctane sulfonamide (F	ND	25	ug/L	2.9		
OSA)						
	PERCENT	RECOVERY				
SURROGATE	RECOVERY	LIMITS		:		
MeFOSA	85	(37 - 130)				



Client Sample ID: E-4

Lot-Sample #: D9K130498-004 Date Sampled: 11/05/09 00:01 Prep Date: 11/16/09 Prep Batch #: 9320505 Dilution Factor: 10	Work Order #: Date Received: Analysis Date: Analysis Time:	11/13/09 12/05/09	Matrix		WATER
	Method:	DEN -LC-00	12		
PARAMETER Perfluorooctanoic Acid Perfluorooctanesulfonate	RESULT 0.13 J 0.22	REPORTING LIMIT 0.20 0.20	UNITS ug/L ug/L	MDL 0.098 0.13	
SURROGATE 13C4 PFOA 13C4 PFOS	PERCENT RECOVERY 115 98	RECOVERY LIMITS (60 - 155) (45 - 130)			
NOTE(S):					

J Estimated result. Result is less than RL.



Client Sample ID: E-4

HPLC

Lot-Sample #...: D9K130498-004 Work Order #...: LPE742AA Matrix..... WATER

 Date
 Sampled...:
 11/05/09
 00:01
 Date Received...:
 11/13/09

 Prep
 Date....:
 11/16/09
 Analysis Date...:
 12/23/09

 Prep
 Batch #...:
 9320505
 Analysis Time...:
 22:57

Dilution Factor: 10

Method..... DEN -LC-0012

PARAMETER	RESULT	REPORTIN LIMIT	G UNITS	MDL	
Perfluorobutanoic acid (PFBA)	0.24	0.20	ug/L	0.098	
Perfluoropentanoic acid (PFPA)	0.41	0.30	ug/L	0.11	
Perfluorohexanoic acid (PFHxA)	0.30	0.20	ug/L	0.029	
Perfluoroheptanoic acid (PFHpA)	ND	0.20	ug/L	0.13	
Perfluorononanoic acid (PFNA)	ND	0.20	uq/L	0.17	
Perfluorodecanoic acid (PFDA)	ND	0.20	ug/L	0.078	
Perfluoroundecanoic acid (PFUn A)	ND	0.20	ug/L	0.069	
Perfluorododecanoic acid (PFDo A)	ND	0.20	ug/L	0.15	
Perfluorotridecanoic acid (PFT riA)	ND	0.20	ug/L	0.18	
<pre>Perfluorotetradecanoic acid (P FTeA)</pre>	ND	0.20	ug/L	0.15	
Perfluorobutane sulfonate (PFB S)	0.95	0.20	ug/L	0.082	
Perfluorohexane sulfonate (PFH xS)	ND:	0.30	ug/L	0.070	
	PERCENT	RECOVERY			
SURROGATE	RECOVERY	LIMITS			
13C4 PFOA	138	(60 - 15	5)		
13C4 PFOS	115	(45 - 13	0)		
13C4 PFBA	112	(36 - 13	0)		
13C2 PFHxA	124	(55 - 13	5)		
1802 PFHxS	114	(61 - 13	0)		
13C5 PFNA	116	(54 - 13	2)		
13C2 PFDA	133 *	(53 - 13	0)	,	
13C2 PFUnA	124	(37 - 13	0)		
	114	(26 - 13			

^{*} Surrogate recovery is outside stated control limits.



Client Sample ID: E-4

Lot-Sample #: D9K130498-004	Work Order #:	LPE741AC	Matri	x:	WATER
Date Sampled: 11/05/09 00:01	Date Received:	11/13/09			
Prep Date: 11/16/09	Analysis Date:	12/01/09			
Prep Batch #: 9320512	Analysis Time:	18:18			
Dilution Factor: 10					
	Method:	DEN -LC-00			
		REPORTING			
PARAMETER	RESULT	LIMIT	UNITS	MDL	
Perfluorooctane sulfonamide (F	ND	0.50	ug/L	0.057	
OSA)					
	PERCENT	RECOVERY		,	
SURROGATE	RECOVERY	LIMITS			
MeFOSA	94	(37 - 130)			

